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Original article

Adhesive luting to hybrid ceramic and resin composite CAD/CAM Blocks: Er:YAG Laser versus chemical etching and micro-abrasion pretreatment.

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Abstract

Purpose: To evaluate the effect of Er:YAG laser on the roughness, surface topography, and bond strength to resin luting cement based on chemical and micro-abrasion pretreatments of different computer-aided design/computer-aided manufacturing materials.

Methods: A polymer-infiltrated-ceramic-network (PICN) material (Vita Enamic, VE), three indirect resin composite (Cerasmart, CS; Shofu HC, SH; Lava Ultimate, LU), and one lithium disilicate ceramic (IPS e.max CAD, EM) blocks were subjected to one of the following pretreatments: no treatment (NC), Er:YAG etching with one of two powers (either 3 or 6 W), hydrofluoric acid (HF) etching, self-etching ceramic primer (ME), or micro-abrasion (MA). The shear bond strength (SBS) of resin luting cement to pretreated materials was tested. Surface roughness was measured via atomic force microscopy, and surface topography was analyzed via scanning electron microscopy. Two-way analysis of variance, Tukey post-hoc test, and Pearson correlation were applied.

Results: Etching EM and VE with HF or the ME resulted in the highest SBS values in their groups ($P < 0.05$). LU, SH, VE, and CS indicated similar SBS values when treated with 3 W, 6 W, and MA. The highest surface roughness (Sa) values were obtained for the LU, CS, and VE groups when treated with 6 W, whereas the lowest Sa values were obtained for CS when treated with the ME and EM when treated with the ME or 3 W. Only SH and CS indicated a significant correlation between surface roughness and bond strength.

Conclusions: Er:YAG laser etching is comparable to micro-abrasion when treating resin composite blocks and may induce fewer surface cracks. HF etching remains the gold standard for the treatment of glass-based ceramics and PICNs.

Keywords: *Luting resin cement, CAD/ CAM ceramics, Hybrid ceramics, Surface treatment, Er:YAG laser*

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1. Introduction

In the past three decades, restorative dentistry has progressed significantly. With the advent of computer-aided design/computer-aided manufacturing (CAD/CAM), dentists are able to fabricate durable dental restorations that exhibit better properties than conventional dental restorations and can be completed in a single visit.[1] Currently, various CAD/CAM ceramic blocks that offer good mechanical properties and uniform material quality are available. The blocks vary in their mechanical strengths from the brittle feldspathic glass-ceramic, to the lithium disilicate glass ceramics, and further to the tough densely-sintered yttrium-stabilized zirconium oxide.[2]

The use of glass-ceramic restorative materials is limited owing to their brittle nature and the abrasive wear induced by glass-based ceramics on opposite dentition.[3] Hence, resin composite-containing blocks were introduced to overcome the increased wear of the antagonist dentition and brittleness associated with glass-ceramic blocks.[4] However, the inferior esthetic properties of resin blocks resulted in the manufacturing of new hybrid formulations, which encompass both the ceramic properties such as high mechanical strength, durability, and color stability, as well as high fracture toughness and low abrasiveness of resins.[5,6] Resin-ceramic CAD/CAM materials can be classified into two categories: polymer-infiltrated-ceramic-network (PICN) materials (also known as “hybrid ceramics”), which comprises a porous ceramic network infused with polymer [7]; or indirect resin composite blocks that exhibit the basic composition of direct composites. Both types of materials are industrially polymerized at high temperatures and pressures and consequently exhibit promising ultrastructure, physical, and mechanical properties, as reported in laboratory studies.[8–10]

It is widely accepted that the appropriate surface pretreatment of indirect restorative materials before luting cementation is critical for improving the adhesion of luting cements to restorations and can consequently increase the longevity of the restorations.[11] However, the surface pretreatment method mainly depends on the chemical

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composition of the material used.

For conventional glass-based ceramic materials, the most typically used method is hydrofluoric acid (HF) etching. Surface pretreatment with HF results in increased surface roughness, thereby increasing the bonding strength between the ceramic and hydrophobic luting cement.[12] Although HF treatment is considered as one of the gold standards mainly for glass ceramics, toxicity issues associated with HF limit its usage.[13] Hence, other etchants that are less toxic than HF have been used in many studies,[14,15] including self-etching ceramic primers. Previously, we demonstrated that the etching efficacy of Monobond Etch and Prime was purely material dependent.[16] Meanwhile, micro-abrasion with alumina particles (sandblasting) is the typically recommended surface treatment method for resin-based blocks.[17] Although sandblasting may provide a clean rough surface for adhesion, the pressure at which blasting occurs has an important role, and using high pressure may induce cracks on the surface and subsurface of the material, thereby reducing the cohesive strength of the material at the surface and subsequently result in failure at the interface.[18]

For indirect resin composite blocks, the absence of a glassy phase renders them resistant to etching by hydrofluoric acid (HF). Hence, alternative methods, such as micro-abrasion and laser ablation pretreatments, have been employed.[19] Various laser systems, such as carbon dioxide (CO_2), neodymium-doped: yttrium, aluminum, garnet (Nd:YAG) or erbium, chromium-doped:yttrium, scandium, gallium, garnet (Er,Cr:YSGG) laser have been utilized in recent studies to improve the surface roughness of hybrid resin ceramics or enhance the repair bond strength of laboratory composite materials.[20,21] Laser surface treatments improved the roughness, thereby enhancing the bond strength between luting resin cement and the fitting surface of the restorations.[22] Er:YAG laser (Erbium: yttrium, aluminum, garnet) is getting popular because of its wide acceptance in clinical dentistry applications, such as carious dentin removal, cavity preparation, surface conditioning, and recently as a surface treatment method for indirect restorations.[23] However, information regarding the effect of hard tissue laser etching, such as Er:YAG laser, on the bonding efficiency of PICN and indirect resin composite materials is scarce.

Hence, in this study, we aim to evaluate the effect of Er:YAG laser pretreatment, in comparison to chemical etching and micro-abrasion, on the surface topography and roughness of PICN and indirect resin composite materials and their bonding efficiency to resin luting cement, by evaluating their shear bond strengths. The null hypothesis is that the surface pretreatment method does not significantly affect the shear bond strength, surface topography, and roughness of the tested materials.

2. Methods and Materials

The materials tested as well as their compositions and manufacturers are shown in Table 1. The study design and pretreatment of samples are illustrated graphically in Figure 1.

2.1. Specimen Preparation

CAD/CAM blocks from each material were used, and were cut transversely to obtain 96 rectangular slabs (12.0 mm × 14.0 mm × 2.0 mm) using a low-speed diamond wheel saw (Isomet 1000, Buehler, Lake Bluff, IL) operating at 500 rpm under cooling water. After ultrasonic cleaning in a bath filled with double-distilled water for 15 min, the specimens were positioned in polyvinyl chloride plastic rings and embedded in epoxy resin (Fastray, Harry J. Bosworth Co., Skokie, IL, USA). The surfaces of the slabs were wet polished with up to 600-grit silicon carbide paper discs (EcoMet 30, Buehler, Lake Bluff, IL) for 1 min. Ten samples from each group (n = 10) were subjected to one of the following pretreatments.

1.No treatment (Negative Control – NC): the specimens did not receive any pretreatment and was primed with a ceramic primer (Mononbond plus, Ivoclar Vivadent) that was applied for 60 s and air dried with a jet of oil-free air for 10 s.

2.Er:YAG laser ablation (Groups 3W and 6W): Laser etching was performed with an Er:YAG laser (Fidelis AT, Fotona Medical Lasers, Ljubljana, Slovenia) operating in a focused mode with a 0.9 mm beam spot of a non-contact handpiece (R02, Fotona) positioned 8 mm from the specimen's surface; pulse duration = 300 µm (short pulse mode - SP), repetition rate = 20 Hz, and water/air spray (80 mL/min water and 40 mL/min air) was performed. The Er:YAG laser power density and energy fluence were 5.97 W/cm^2 and 29,856 J/cm^2, respectively. Laser irradiation was performed with one of two different laser power settings: a) 150 mJ and b) 300 mJ, with power outputs of 3 W (Group 3W) and 6 W (Group 6W), respectively.
The specimens were rinsed off with water/air spray for 30 s and dried with a strong jet of oil-free air for 10 s; subsequently, Mononbond Plus was applied for 60 s and then air dried.

3.Hydrofluoric acid (Group HF): A 4.5% HF was applied for 60 s except for the IPS e.max CAD (EM) samples, which were etched for only 20 s according to the manufacturer's instructions except for IPS e.max CAD (EM). All the samples were washed with water/air spray for 30 s and then air dried for 10 s. Mononbond plus was applied for 60 s and then air dried.

4.Monobond Etch & Prime (Group ME): A self-etching glass-ceramic primer (Monobond Etch & Prime, Ivoclar Vivadent) was applied on the surface using a microbrush, agitated into the surface for 20 s, and then allowed to react for another 40 s; subsequently, it was thoroughly rinsed off with water/air spray for 30 s and dried with a strong jet of oil-free air for 10 s.

5.Micro-abrasion (Group MA): air abrasion with 25 µm alumina particles (Cobra Abrasive, Renfert GmbH, Hilzingen, Germany) was performed in a sandblaster (Basic Eco, Renfertat GmbH, Hilzingen, Germany) at a pressure of 200 kPa from a distance of approximately 10 mm for 14 s. The specimens were ultrasonically cleaned in a distilled water bath for 30 s and then rinsed with water/air spray for 30 s. The samples were dried with a strong jet of oil-free air for 10 s, and Mononbond Plus was applied for 60 s and then air dried.

After surface treatment and priming, a dual-cure resin cement (Multilink –N Automix, Ivoclar Vivadent) base and catalyst pastes were mixed in an automixing tip and then applied to the ceramic surface. A resin composite cylinder, 2.0 mm in diameter and 2.0 mm in height, was placed, and a special metal clip was used to stabilize the resin composite cylinder during the cementation. Excess cement was removed using a microbrush, and the cement was light cured for 20 s using light-emitting diode (LED) curing unit (Elipar DeepCure, 3M ESPE) with an output of 1470 mW/cm^2. The intensity was verified in every 10 samples using a digital radiometer (Cure Rite, Dentsply, Milford, DE, USA) to ensure uniform curing. Each specimen was examined using magnifying loupes to identify specimens containing possible defects (bubbles or cracks in resin composite or flow of resin cement beyond the limits of the bonding area).

2.2. Shear Bond Strength (SBS) Testing

The samples were stored in distilled water at 37 °C for 24 h and thermocycled between 5 °C and 55 °C for 5,000 cycles with 30 s dwell times (THE-1100, SD Mechatronik, Germany) before being tested for their SBS using a table-top shear bond strength tester (Bisco Inc., Schaumburg, IL, USA). The metal blade of the machine was passively placed on top of the sample base surface (the bonding substrate), and the distance between the blade and sample bases was set to 0.1±0.05 mm, measured using a feeler gauge (Elora–Werkzeugfabrik GmbH, Remscheid, Germany). To exclude the presence of any frictional forces, the passive movement of the blade was confirmed before testing each sample (i.e., no forces were recorded by the load cell during the advancement of the blade until the blade touched the sample). The metal blade was adjusted such that the semicircular notch of the blade

Table 1. The materials tested in this study.

Material	Code		Manufacturer	Composition	Lot #
Vita Enamic	VE	Polymer-infiltrated ceramic network (PICN)	Vita Zahnfabrik H. Rauter GmbH, Bad Säckingen, Germany	Bis-GMA, UDMA, Bis-EMA, TEGDMA, polymer network (14 wt%) SiO_2, Al_2O_3, Na_2O, K_2O, B_2O_3, ZrO_2, CaO ceramic network (86 wt%)	38630
Cerasmart	CS	Resin Composite	GC Dental Products, Leuven, Belgium	BisMEPP, UDMA, DMA (29 wt%) SiO_2 and B_2O_3 glass nanofillers (71 wt%).	1610051
Shofu Block HC	SH	Resin Composite	Shofu Dental, Kyoto, Japan	UDMA, TEGDMA (39 wt%) SiO_2, ZrO_2 fillers (61 wt%)	031501
Lava Ultimate	LU	Resin Composite	3M ESPE, St. Paul, MN, USA	BisGMA, UDMA, BisEMA, TEGDMA (20 wt%) SiO_2, ZrO_2 fillers (80 wt%)	N333039
IPS e.max CAD,	EM	Lithium disilicate glass-ceramic	Ivoclar Vivadent, Schaan, Liechtenstein	Lithium disilicate reinforced glass-ceramic (SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, Al_2O_3, MgO, and Coloring oxides)	U26002
Monobond Etch & Prime	ME	Self-etching glass-ceramic primer	Ivoclar Vivadent, Schaan, Liechtenstein	Tetrabutylammonium dihydrogen trifluoride, methacrylated phosphoric acid ester, trimethoxysilylpropyl methacrylate, alcohol, and water.	U12508
IPS Ceramic Etching Gel	HF	Ceramic Etching	Ivoclar Vivadent, Schaan, Liechtenstein	4.5% hydrofluoric acid	U01182
Monobond Plus,		Glass-ceramic primer	Ivoclar Vivadent, Schaan, Liechtenstein	Ethanol, 3-trimethoxysilylpropyl methacrylate, 10-MDP, Sulfide methacrylate	U25466
Multilink Automix		Dual Cure Luting resin cement	Ivoclar Vivadent, Schaan, Liechtenstein	Dimethacrylates, HEMA, t-amine, silicon dioxide filler, ytterbium trifluoride, catalysts, stabilizers, pigments, dibenzoyl peroxide	U02278

*Bis-GMA (2,2-bis[4-(2-hydroxy-3-methacryloxypropoxy)phenyl]propane), UDMA (urethane dimethacrylate), Bis-EMA (2,2-Bis[4-methacryloxypolyethoxyphenyl)propane]), TEGDMA (Triethylene glycol dimethacrylate), 10-MDP(10-methacryloyloxydecyl dihydrogen phosphate), HEMA (2-hydroxyethyl methacrylate).

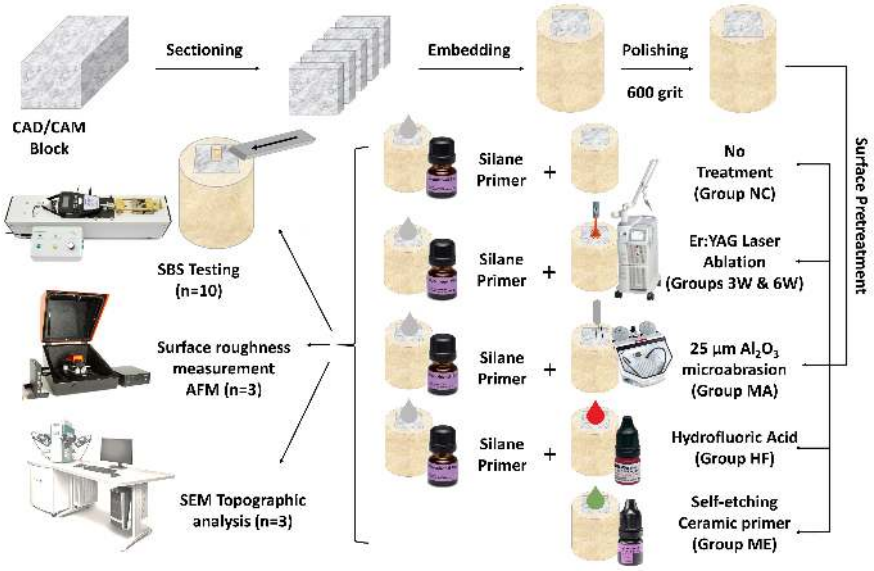

Fig. 1. Schematic illustration of sample preparation and pretreatments of tested groups.

was passively encircling the resin cylinder. The metal blade was advanced to apply shear forces at the resin–ceramic interface and operated at a crosshead speed of 1.0 mm/min until complete failure and debonding. The force at failure, recorded in Newtons, was divided by the surface area (mm^2) to calculate the SBS in millipascal using the following formula:

SBS (MPa) = Force (N) /Area (mm^2)

The debonded specimens were analyzed under a stereomicroscope to determine the failure mode, which was classified as one of the following: the adhesive failure between the resin cement and ceramic (A), mixed failure (M), cohesive failure in resin cement (CR), or cohesive failure in ceramic (CC).

2.3. Surface roughness analysis

To analyze the surface roughness, 18 samples from each material tested were further polished with a series of silicon carbide paper discs (800, 1000, and 1200 grit), then divided equally into 6 groups (n=3) accourding to the different surface treatments. Subsequently, they were evaluated via atomic force microscopy (AFM) (Flex-Axiom, Nanosurf AG, Liestal, Switzerland) performed in a non-contact mode using an AFM cantilever with magneto-resistive sensors integrated within an NCLR tip (7 µm thickness, 225 µm length, and 38 µm width) under a force constant of 48 N/m. Measurements from four different areas were collected using a standardized rectangular spot (25 µm × 25 µm). The tip-specimen distance was maintained throughout the test. The AFM micrographs were analyzed using microscopy data analysis software (C3000 control software, version 3.7.2.8, Nanosurf AG, Liestal, Switzerland). The average surface roughness (Sa), peak height (highest value - Sp), and valley depth (lowest value - Sv) of the pretreated CAD/CAM materials were measured and expressed as a numeric value in nanometers.

2.4. Surface Topography Examination

3 specimens from each group were polished and treated as explained for surface roughness analysis, air dried for 60 s and stored in a desiccator containing dehydrated silica gel for 24 h. The dehydrated samples were then mounted on aluminum stubs with double-sided adhesive carbon tape. The specimens were coated with gold-palladium in a sputter-coater (Polaron E- 5100 Sputter Coater, Polaron Equipment Ltd, Watford, UK) at 20 mA for 90 s. Micrographs of the prepared samples were obtained at different magnifications up to 2000X using a low-vacuum scanning electron microscope (JSM 5310LV, JEOL Inc., Tokyo, Japan) operating at an accelerating voltage of 20 kV and a working distance of 12.0–17.0 mm.

2.5. Statistical analysis

A statistical analysis of the results for the SBS and surface roughness parameters was performed via a two-way analysis of variance (CAD/CAM material and surface pretreatment method) to determine the effect of these major factors on the SBS and surface roughness. Tukey's post-hoc test was used to detect pair-wise differences among experimental groups. A 95% confidence level was applied for all statistical tests (a = 0.05), and the power of the analysis with a = 0.05 was 0.90. The statistical analysis was performed using SPSS software (Version 15.0, SPSS Inc., Chicago, IL, USA). The correlation between the mean SBS and roughness of each tested material was then computed using a two-tailed Pearson's correlation analysis.

3. Results

The mean SBS values are summarized in Table 2. During the SBS testing of all groups, no pre-testing failures occurred. The SBS results were affected significantly by both variables, the pretreatment method, and the CAD/CAM block substrate type ($p < 0.05$).

Compared with all other treatment methods, both VE and EM exhibited significantly higher mean SBS values ($p < 0.05$) when they were treated with HF or ME, with the HF-treated EM group attaining the highest value (40.18±9.30 MPa), followed by the ME-treated EM group (26.66 ± 5.9 MPa) and the HF-treated VE group (26.40±5.8 MPa). The treatment of the EM group with 3 W laser resulted in the lowest SBS value (3.92±1.52 MPa) among all the groups.

Meanwhile, no significant difference ($p > 0.05$) was observed in VE, LU, SH, and CS groups when they were treated with either 3W, 6W, or MA. The SBS of the MA-treated LU group was significantly higher than those of the HF- and ME-treated groups ($p < 0.05$), and the SBS of the 3W and 6W-treated SH were significantly higher than those of the HF and ME-treated groups ($p < 0.05$). Whereas the 3W, 6W and MA-treated CS groups exhibited bond strengths that did not differ significantly from that of the HF-treated group ($p > 0.05$).

The types of surface treatment and material tested affected the failure mode of the debonded specimens of the different experimental groups (Table 3). For VE and EM, the highest percentage of adhesive failure was associated with laser pretreatment, and this percentage decreased when they were treated with MA, HF, or ME. By contrast, for the resin composite materials (CS, SH, and LU), the most significant adhesive failures occurred when the samples were treated with either HF or ME. A high percentage of cohesive failure in the CAD/CAM materials was observed when VE was treated with either the ME or HF.

The AFM surface roughness results are listed in Table 4 and presented as three-dimensional micrographs in Figures 2–6. Pretreatment with HF or the ME induced lower mean Sa compared with MA and laser pretreatment for all tested materials except EM, which showed the highest mean Sa value when treated with MA and HF surface etching. HF etching exhibited significantly higher Sa compared with ME treatment.

Representative SEM micrographs of the tested groups are presented in Figures 2–6. Compared with the NC groups, both types of laser pretreatments resulted in intense irregularities and pores in all materials tested except for EM, which remained relatively smooth after treatment with 3W (Figure 6B) and revealed only slight irregularities when treated with 6W (Figure 6C). In general, the 6W pretreatment groups (Figures 2–6 C) exhibited more irregularities and surface roughness than the 3W groups (Figures 2–6 B). By contrast, HF treatment (Figures 2–6 E) resulted in rough surfaces with micro-irregularities and micropores on the treated surfaces of all materials tested except for CS (Figure 4E), which showed a comparatively smoother surface. In comparison to HF etching, ME etching generated a less pronounced etching pattern in all the tested groups, and this mild etching pattern was particularly obvious in EM and VE (Figures 5F and 6F), indicating a partial dissolution of the glassy matrix, fewer number of pores, and smoother surfaces compared with HF etching. The SEM micrographs of the micro-abraded resin composite blocks (Figures 2–6D) show a substantially rough surface with deep and wide valleys (> 20 μm), which correspond to the micro-abrasive particles used for MA. Multiple surface cracks (white arrows) were observed, particularly after the MA treatment of LU, SH, VE, and EM (Figures 2D, 3D, 5D, and 6D, respectively) and with the laser etching of SH (Figure 3B). Other characteristic features in the SEM analysis include cracks and holes in the spherical particles of untreated and 6 W laser-etched SH (Figures 3A and 3C, respectively).

No significant correlation was discovered between surface roughness and bond strength for any of the tested materials except for SH and CS, which showed relatively higher Pearson correlation coefficients ($R^2 = 0.664$ and 0.7102, respectively). Meanwhile, this correlation was relatively weak in the cases of LU, EM, and VE ($R^2 = 0.442$, 0.109, and 0.028, respectively).

4. Discussion

In the current study, a lithium disilicate glass-ceramic, a hybrid ceramic (PICN), and resin composite-based CAD/CAM materials were studied, and multiple surface treatment methods were evaluated. The results revealed that the effect of surface treatments on the surface roughness and SBS of the CAD/CAM materials tested were material dependent. Compared to the resin-based materials, EM and VE showed significantly higher mean SBS values when treated with HF and ME than those obtained with MA and laser treatments, whereas SH and LU showed significantly higher SBS values when sandblasted or laser treated. For CS, the SBS did not differ significantly among the treatment methods. Additionally, SEM and AFM analysis showed that different treatments resulted in different surface patterns and degrees of surface roughness. These data support the rejection of the null hypothesis.

A fully crystallized EM is composed of 70% fine-grain lithium disilicate crystals ($Li_2Si_2O_5$) embedded in a glassy matrix, whereas VE is composed of a porous feldspar ceramic network enriched with aluminum and zirconium oxide, as well as infiltrated with a monomer mixture, which is then cured to form a polymer network within the ceramic network. [7, 8] Consequently, VE behaves more like a feldspathic ceramic than a resin composite-based material. HF etching results in changes in the surface microstructure of EM due to the preferential dissolution of the amorphous glassy matrix of porcelain, which is selectively removed, thereby exposing the crystalline lithium structure and creating a rough surface; this is a more appropriate microstructure for bonding (Figure 6E). A similar effect was observed when etching VE, where the feldspathic matrix was selectively removed, thereby creating significant micro-irregularities and exposing the resin network (Figure 5E).[9] The subsequent application of a silane coupling agent to the etched ceramic surface provides a chemical covalent hydrogen bond via the condensation of the silanol group of activated silane with the hydroxylated silica groups of the acid-etched ceramics; the covalent hydrogen bond was further stabilized via

Table 2. Mean Shear Bond Strength Values (MPa ± standard deviation) of Lava Ultimate (LU), Shofu HC (SH), Cerasmart (CS), Vita Enamic (VE), and IPS e.max CAD (EM), after no treatment (NC) or pretreatment with hydrofluoric acid (HF), self-etching ceramic primer (ME), Er:YAG etching with either (3W) or (6W) power, or microabrasion (MA)..

	LU	SH	CS	VE	EM
NC	2.32 1.06c, A	2.74±0.9c,A	0.86 1.6c,A	3.53±1.7d,A	3.62±2.0d, A
HF	13.47±5.2a,A	12.02±3.3a,A	16.38±6.3ab,A	26.40±5.8a,B	40.18±9.3a,C
ME	12.76±6.2a,A	10.07±2.2a,A	10.34±2.0a,A	21.41±6.9a,B	26.66±5.9b.B
3W	17.77±2.8ab,AB	20.64±3.1b,A	15.72±6.2ab,AB	14.73±4.2c,B	3.92±1.5d,C
6W	18.23±2.1ab,AB	23.25±1.4b,A	20.02±5.9b,A	16.62±7.3bc,AB	13.24±3.8c,B
MA	21.98±7.4b,A	15.56±4.0ab,B	20.15±3.7b,AB	17.74±6.7bc,AB	18.60±2.2c,AB

*Within a column, the same Lower-case superscript letters show mean values with no statistically significant difference ($p > 0.05$).
*Within a raw, the same Upper-case superscript letters show mean values with no statistically significant difference ($p > 0.05$).

Table 3. Mode of failure (%) following the shear bond strength test of Lava Ultimate (LU), Shofu HC (SH), Cerasmart (CS), Vita Enamic (VE), and IPS e.max CAD (EM), after no treatment (NC) or pretreatment with hydrofluoric acid (HF), self-etching ceramic primer (ME), Er:YAG etching with either (3W) or (6W) power, or microabrasion (MA).

Material	Pretreatment	Mode of Failure %			
		A	M	CR	CC
LU	NC	100	0	0	0
	HF	70	30	0	0
	ME	90	10	0	0
	3W	0	40	60	0
	6W	0	80	20	0
	MA	0	100	0	0
SH	NC	100	0	0	0
	HF	80	20	0	0
	ME	100	0	0	0
	3W	0	100	0	0
	6W	0	80	20	0
	MA	0	80	20	0
CS	NC	100	0	0	0
	HF	40	60	0	0
	ME	90	10	0	0
	3W	10	90	0	0
	6W	20	60	20	0
	MA	10	90	0	0
VE	NC	100	0	0	0
	HF	0	20	0	80
	ME	10	10	0	80
	3W	90	10	0	0
	6W	70	10	10	10
	MA	10	70	10	10
EM	NC	100	0	0	0
	HF	10	20	0	70
	ME	10	50	0	40
	3W	80	20	0	0
	6W	100	0	0	0
	MA	10	80	10	0

*A = Adhesive; M = Mixed; CR = Cohesive in resin cement; CC = Cohesive in ceramic

silanol intermolecular condensation. Meanwhile, the methacrylate group of the silane primer contributed to the chemical bonding to the optional methacrylate-based resin adhesive or directly to the resin cement.[24]

The ME, which has recently been introduced for the surface pretreatment of indirect esthetic restorations, contains ≤ 10% tetrabutylammonium dihydrogen trifluoride and silane (trimethoxysilylpropyl methacrylate). The ME is applied in one step for 60 s, where the polyfluoride acidic salt partially dissolves the glassy phase of the ceramic surface, and a thin layer of silane remains on the ceramic surface after rinsing the primer with air/water spray. Compared to conventional HF and silane treatment, the new simplified technique requires a shorter time and aims to eliminate the toxic potential of HF and minimize the technique sensitivity of the process. [7]However, after SEM and AFM analyses, it was evident that the ME was less aggressive than HF and hence yielded a weaker etching pattern on the ceramic surface (Figures 2–6F); these findings agreed well with those of many previous studies.[16,25,26] Although the ME is less acidic than HF (pH = 2.0 for HF, and 3.8 for ME),[27] the weaker etching capacity of the ME cannot be attributed to the difference in acidity. Tian et al. explained that the porcelain dissolution process does not depend on the acidity of HF or the ME; instead, it depends on the reaction of fluoride with silicon dioxide to form silicon fluoride.[28] Hence, the different etching effects of HF and the ME can

Table 4. Mean average surface roughness (Sa), peak height (highest value - Sp) and valley depth (lowest value - Sv) of Lava Ultimate (LU), Shofu HC (SH), Cerasmart (CS), Vita Enamic (VE), IPS e.max CAD (EM), after no treatment (NC) or pretreatment with hydrofluoric acid (HF), self-etching ceramic primer (ME), Er:YAG etching with either (3W) or (6W) power, or microabrasion (MA).

Material	Pretreatment	Sa (nm)	Sp (nm)	Sv (nm)
LU	NC	39.852	160.38	-332.46
	HF	129.54	1068.20	-900.01
	ME	91.40	418.49	-903.36
	3W	784.99	366.26	-3.3223
	6W	830.87	1487.40	-2013.20
	MA	475.53	2275.70	-1837.90
SH	NC	75.89	318.23	-483.38
	HF	83.37	483.82	-388.49
	ME	63.20	451.92	-371.91
	3W	563.03	2593.30	-1778.40
	6W	548.24	3286.80	-2915.30
	MA	615.06	385.30	-640.30
CS	NC	42.81	403.44	-266.66
	HF	55.45	234.61	-408.72
	ME	27.18	167.36	-245.61
	3W	458.92	1121.20	-1289.20
	6W	856.36	3271.60	-3847.80
	MA	756.57	2726.00	-3037.00
VE	NC	56.697	614.28	-408.78
	HF	443.47	3027.80	-3681.00
	ME	161.38	866.26	-675.70
	3W	458.92	2165.50	-3095.20
	6W	870.06	5155.00	-3003.20
	MA	313.76	1312.40	-1180.20
EM	NC	69.63	272.33	-397.15
	HF	163.71	1601.30	-1779.70
	ME	33.14	188.83	-371.28
	3W	37.49	203.82	-149.16
	6W	58.23	508.08	-255.45
	MA	430.11	2485.90	-1454.00

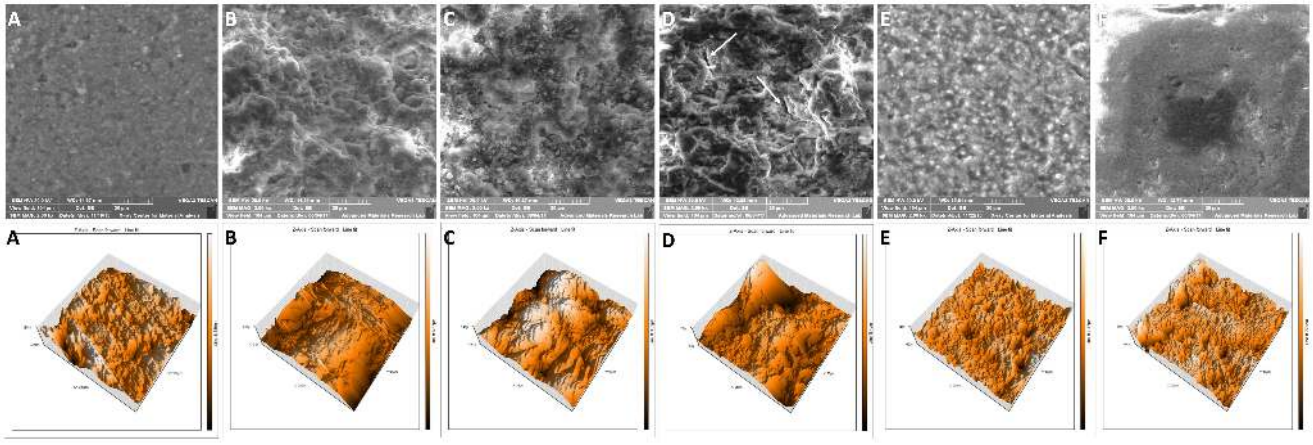

Fig. 2. SEM (top) and AFM (bottom) micrographs of Lava Ultimate (LU) after different pretreatments. (A) no treatment, (B) Er:YAG etching with 3W power, (C) Er:YAG etching with 6W power, (D) microabrasion, (E) hydrofluoric acid, or (F) Monobond Etch and Prime. The white arrows indicate cracks.

be attributed to the variation in the reactivity of the available fluoride within each material.

In the present study, the bond strengths of HF and the ME did not differ significantly in any of the material groups tested except for EM, where HF treatment resulted in a significantly higher bond strength. Previous studies showed that the ME was efficient in conditioning vitreous ceramics, presenting bond strengths that were comparable to those of the conventional HF treatment.[25] In one study, the ME showed a more stable bond after aging.[26] However, no aging was performed in most of these studies. Other studies concluded that conventional treatment with HF followed by silanization remained as the gold standard; furthermore, this treatment yielded higher mean

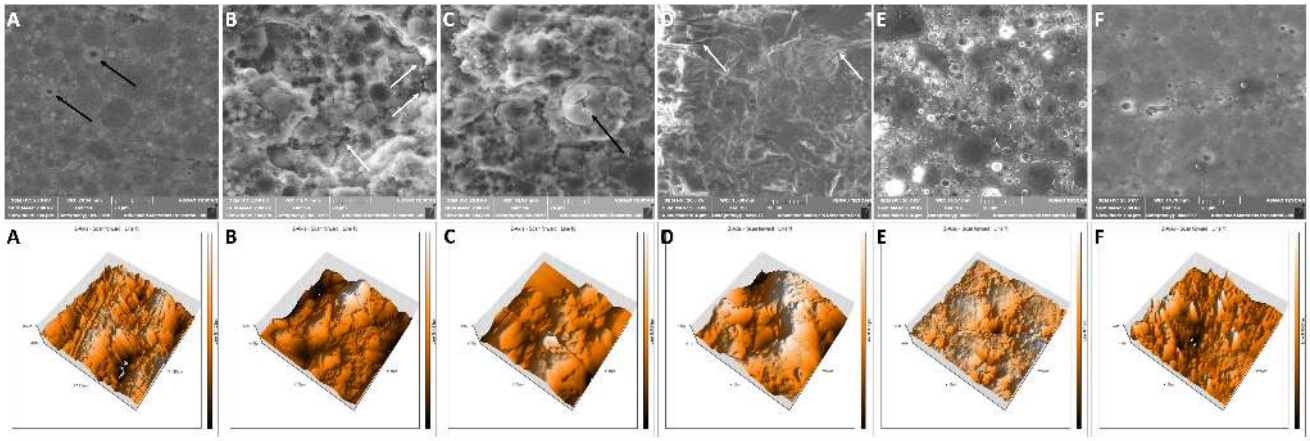

Fig. 3. SEM (top) and AFM (bottom) micrographs of Shofu HC (SH) Blocks after different pretreatments. (A) no treatment, (B) Er:YAG etching with 3W power, (C) Er:YAG etching with 6W power, (D) microabrasion, (E) hydrofluoric acid, or (F) Monobond Etch and Prime. The white arrows indicate cracks, and black arrows indicate holes in the spherical fillers.

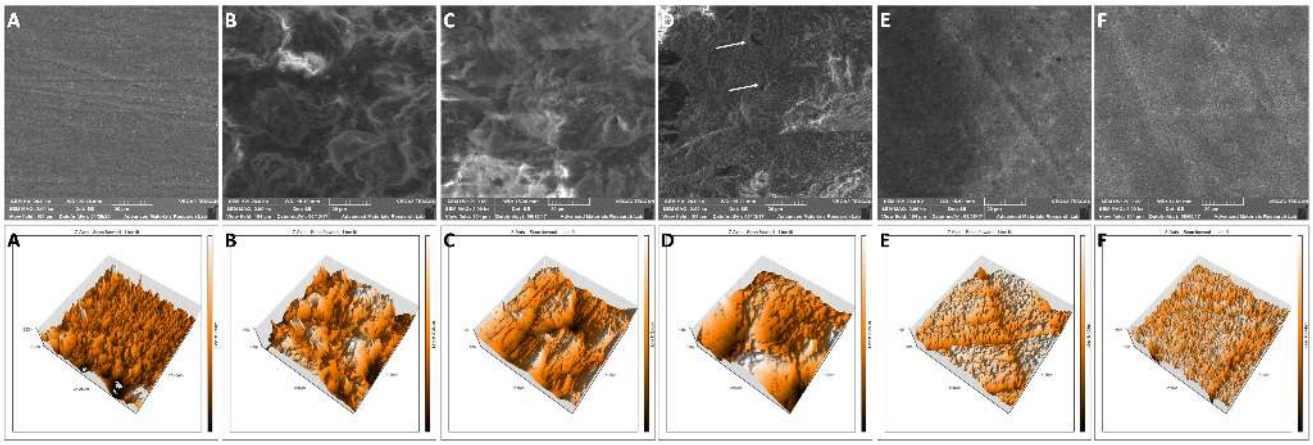

Fig. 4. SEM (top) and AFM (bottom) micrographs of Cerasmart (CS) after different pretreatments. (A) no treatment, (B) Er:YAG etching with 3W power, (C) Er:YAG etching with 6W power, (D) microabrasion, (E) hydrofluoric acid, or (F) Monobond Etch and Prime. The white arrows indicate cracks.

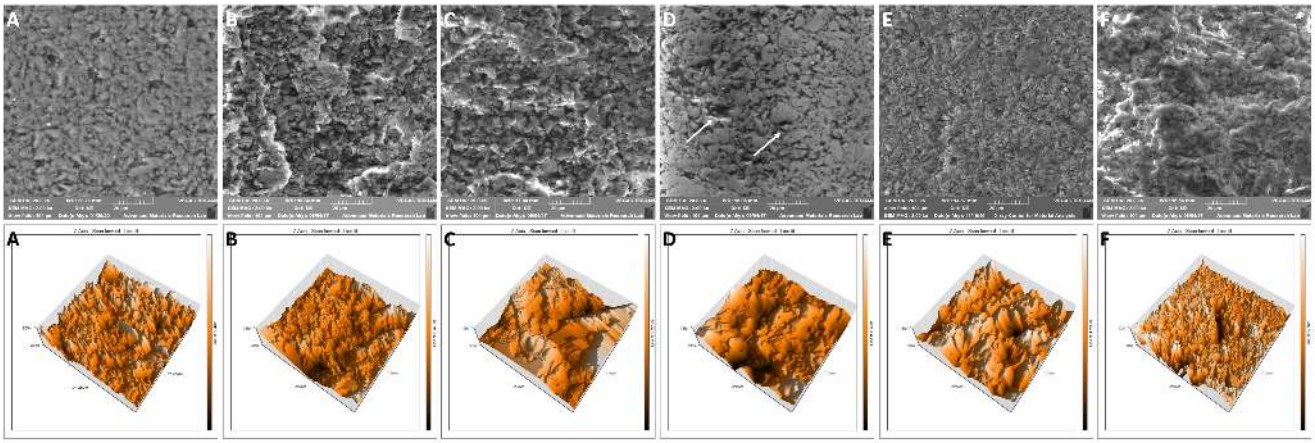

Fig. 5. SEM (top) and AFM (bottom) micrographs of Vita Enamic (VE) after different pretreatments. (A) no treatment, (B) Er:YAG etching with 3W power, (C) Er:YAG etching with 6W power, (D) microabrasion, (E) hydrofluoric acid, or (F) Monobond Etch and Prime. The white arrows indicate cracks.

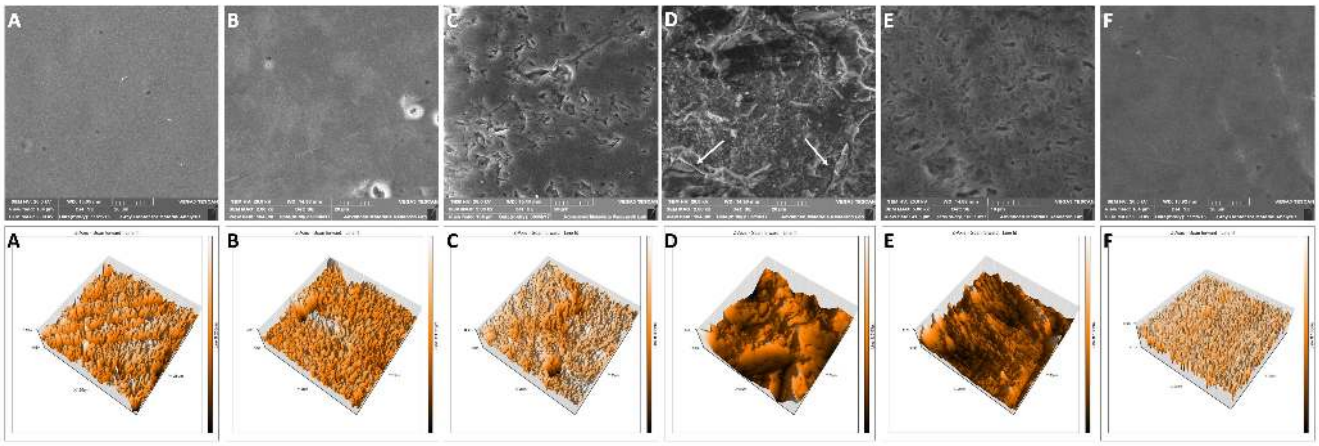

Fig. 6. SEM (top) and AFM (bottom) micrographs of IPS E.max CAD (EM) after different pretreatments. (A) no treatment, (B) Er:YAG etching with 3W power, (C) Er:YAG etching with 6W power, (D) microabrasion, (E) hydrofluoric acid, or (F) Monobond Etch and Prime. The white arrows indicate cracks.

bond strength values when applied on lithium disilicate and feldspathic ceramics, compared with using the ME on lithium disilicate ceramic surfaces, which resulted in significantly lower SBS values, consistent with the findings of our study.[15,29]

On the contrary, LU, CS, and SH are fabricated from a resin-based matrix enforced with ceramic fillers; this may explain the necessity of performing mechanical roughening on the surface rather than HF etching. A study by Soares et al. attributed the low bond strength of resin cement to HF-etched indirect resin composite to the dissolution of the glass filler particles and the softening of the resin matrix; furthermore, they reported that the application of silane after HF etching did not improve the bond strength because of the absence of glass filler particles.[30] For various indirect resin composite-based CAD/CAM materials, which are commercially known as resin nano-ceramic blocks, such as LU, SH, or CS, the majority of manufacturers recommend the blasting of the intaglio surface of the restoration before silanization.[8] Sandblasting has been shown to improve bond strength by removing the smear layer created after milling, exposing a fresh surface that is free of contaminants and providing enhancedmicromechanical retention to the cement.[31–33] Sandblasting exposes the filler particles in the composite-based blocks, thereby enhancing the subsequent bonding between these inorganic fillers and the silanol groups of the silane primer by the formation of siloxane bonds, as discussed previously. Meanwhile, the vinyl group of the silane primer contributes to the adhesion to the organic matrix of resin-based materials.[24]

In the current study, MA treatment of the resin- based blocks yielded higher SBS than HF etched resin-based surfaces. Several studies evaluated the effect of the pretreatment strategy of LU, SH, and CS on bonding and similarly reported that for a higher bond strength, the restorations should be air abraded and pretreated using a resin primer containing methyl-methacrylate.[34,35]

The micro-abraded block surfaces of all the investigated materials (Figures 2-6 D), revealed an irregular surface with cracks and some deeply dented parts under the SEM. Similar to our findings, Yoshihara et al. showed that although sandblasting resin composites and hybrid ceramic CAD/CAM blocks increased the surface roughness and resulted in an irregular surface with some filler exposure, it also caused surface and subsurface cracks measuring 1–10 μm. These cracks can be detected inside the resin matrix and at the interface between the resin matrix and filler particle.[18] Other investigators recommended that composite blocks should be sandblasted with a small grain size abrasives (≤ 50 μm) and a reduced pressure of 100–200 kPa, which is lower than the pressure typically recommended for ceramic and metal restorations.[36,37] In the present study, although the sandblasting pressure applied was 200 kPa and the particle size was 25 μm, substantial cracks were still detected after MA treatment on the surfaces of LU, SH, and EM.

An alternative surface treatment method induced by Er:YAG laser irradiation was investigated in this study. During laser treatment, considerable local temperature changes can create internal pressures that can damage the materials; therefore, the appropriate laser parameters must be used to avoid any deleterious effects during laser treatment.[38] In the current study, laser etching was performed with an Er:YAG laser operating at two different laser power settings: 150 and 300 mJ, with a total energy output of 3 and 6W, respectively.

In the present study, the HF and the ME surface treatment of EM resulted in a significantly higher SBS compared with Er:YAG laser irradiation. Both laser settings yielded extremely low mean SBS values that did not exceed 14 MPa. In comparison, the mean SBS values reached 40 MPa when the conventional HF followed by silanization treatment were applied. These findings are in contrast to those reported by Gökçe et al., who concluded that Er:YAG laser with a power setting of 300 mJ exhibited higher SBS values than HF etching when applied to an Empress 2 surface (lithia-based ceramic), indicating that laser etching can be used as an alternative to HF/silane surface treatment.[39] The disagreement may be due to the different settings of the laser operation or differences in the resin cement used. The authors explained that laser irradiation could cause a heat-damaged layer, which might be poorly attached to the inner surface of the substrate while having the outer surface of the substrate strongly bonded to the silane and luting agents. Therefore, the low SBS values of Er:YAG-treated EM and VE in our study is attributable to the fragmentation of the ceramic crystals.

By contrast, both the 3 and 6 W laser irradiations were effective for pretreating LU, SH, and CS, with similar or higher SBS values compared with HF etching techniques. The effect of Er:YAG laser on the ablation of composite resins or the effect of laser treatment on the repair bond strength of laboratory composites has been evaluated in some studies.[20,21] The results of the current study partially agreed with those reported by Cengiz-Yanardag et al., who investigated the pretreatment of hybrid ceramic blocks with Er,Cr:YSGG laser and reported comparable results of high-energy laser treatment to those of sandblasting, except for VE, which exhibited lower bond strength when sandblasted.[24] It has been reported that during composite resin ablation, explosive vaporization occurred at approximately 300 °C, followed by hydrodynamic ejection, which caused instantaneous melting in less than a picosecond. The change in volume of the material upon melting created large expansion forces that resulted in micro-explosions within the ablated surface.[21]

With respect to AFM analysis and SEM topographical analysis, although laser pretreatment and MA revealed significantly rougher surfaces in the PICN and resin composite materials, laser pretreatment induced fewer cracks and destruction in the microstructure of the materials' surfaces compared with MA. The increase in laser power from 150 to 300 mJ did not significantly affect the roughness of LU or SH, whereas the difference was significant for CS and VE.

A weak correlation between the surface roughness and SBS values of EM and VE was revealed. These results indicate that the two materials did not depend mainly on the micromechanical roughness in their adhesion but rather on the chemical adhesion between the ceramic crystals and the silane primer. By contrast, a relatively strong correlation between surface roughness and SBS was calculated for SH and CS, which may indicate the significant effect of surface roughness on the bond strength of these materials.

The results revealed a strong association between the failure mode and the types of surface treatment and material. The highest percentage of adhesive failure was associated with the laser pretreatment for EM, which can be explained by the absence of micro-porosities created. However, a high percentage of adhesive failure, associated with low mean SBS values, was also revealed by the VE treated with laser, even though AFM and SEM analyses showed rough areas with pores; this effect may be attributed to the dissociation of the ceramic network by laser energy, which weakened the bond to the adhesive system. On the contrary, LU, SH, and CS indicated the highest percentage of adhesive failure when treated with either HF or the ME. A high percentage of cohesive failure in the material was observed when VE was treated with HF or the ME. These results are attributable to the difference in the microstructure and its effect on the intrinsic strength of the materials.

One limitation of this study is that the specimens were prepared using a slow-speed diamond saw, followed by polishing with silicon carbide paper discs, which may produce surfaces that are not representative of the surfaces produced by CAD/CAM milling. Moreover, only the thermal fatigue of the samples (5000 thermal cycles) was investigated in the current study. Testing the bond strength after increasing the aging cycles or after a combination of thermomechanical aging can provide a clearer insight into the long-term performance of the tested materials following each pretreatment method. Another limitation is the use of only one adhesive luting resin cement. Therefore, the effect of the pretreatment method in combination with different types of cement, ceramic, and resin materials on the SBS longevity may be investigated in future studies.

5. Conclusion

Within the limitations of the current study, we conclude that the effectiveness of the pretreatment method is mainly material dependent. Chemical etching with HF or self-etching primers is more effective for glass-based ceramics such as lithium disilicate or PICN materials. By contrast, mechanical roughening by micro-abrasion or laser ablation is more effective for resin composite materials. Compared to sandblasting, the erbium laser pretreatment of resin composite resulted in comparable or superior bond strengths to the resin cement, while preserving the resin-based CAD-CAM material microstructure from damage and cracking.

Conflict of interest

The authors of this manuscript certify that they have NO affiliations with or involvement in any organization or entity with any financial interest, or non-financial interest in the subject matter or materials discussed in this manuscript.

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